In-Process Cure Monitoring of Composites via Fiber-Optic Fluorescence

Final Report

C.S.P. Sung and N.H. Sung

October 23, 1992

U.S. Army Research Office
26571-MS
DAAL03-89-K-0081

Institute of Materials Science
University of Connecticut
Storrs, CT 06269-3136

and

Department of Chemical Engineering
Tufts University
Medford, MA 02155

REPORT DOCUMENTATION PAGE

Form Approved
OMB No. 0704-0188

1. AGENCY USE ONLY (Leave blank)	2. REPORT DATE	3. REPORT TYPE AND DATES COVERED
	October 23, 1992	Final Report 4/89-7/92

4. TITLE AND SUBTITLE
In-Process Cure Monitoring of Composites via Fiber-Optic Fluorescence

5. FUNDING NUMBERS
DAAL03-89-K-0081

6. AUTHOR(S)
C.S.P. Sung and N.H. Sung

7. PERFORMING ORGANIZATION NAME(S) AND ADDRESS(ES)
Institute of Materials Science & Dept. of Chemical Eng.
University of Connecticut Tufts University
Storrs, CT 06269-3136 Medford, MA 02153

8. PERFORMING ORGANIZATION REPORT NUMBER

9. SPONSORING/MONITORING AGENCY NAME(S) AND ADDRESS(ES)
U. S. Army Research Office
P. O. Box 12211
Research Triangle Park, NC 27709-2211

10. SPONSORING/MONITORING AGENCY REPORT NUMBER
ARO 26571.4-MS

11. SUPPLEMENTARY NOTES
The view, opinions and/or findings contained in this report are those of the author(s) and should not be construed as an official Department of the Army position, policy, or decision, unless so designated by other documentation.

12a. DISTRIBUTION/AVAILABILITY STATEMENT
Approved for public release; distribution unlimited.

12b. DISTRIBUTION CODE

13. ABSTRACT (Maximum 200 words)

The objective of the research under this contract were to develop in-situ cure monitoring technique based on intrinsic and extrinsic chemical sensors using fiber-optic fluorescence method for epoxy and polyimide and to use such a fiber-optic method to investigate fiber surface effects on epoxy structure and cure kinetics.

As an extrinsic cure sensor, p,p´-diamino azobenzene (DAA) shows sensitive fluorescence intensity enhancement as a result of reaction with epoxide. It has been implemented for in-situ cure monitoring of bulk epoxy cured with diaminodiphenyl dulfone (DDS) at high cure temperatures (140-180°C) using fiber optic fluorescence instrument, using an internal standard dye to calibrate the intensity.

The intrinsic fluorescence excitation spectra of DDS was found to show sensitive spectral shifts with cure. This result has also been implemented with real composites (carbon or glass fiber reinforced prepregs) in actual cure environment by fiber optic fluorescence instrument, by taking into account the effect of temperature on the spectral position.

14. SUBJECT TERMS
Cure Monitoring, Diamine Cured Epoxy, Composites, Fiber-Optic Fluorescence, Polyimides, Extrinsic and Intrinsic Cure Sensor, In-Process Monitoring

15. NUMBER OF PAGES
2

16. PRICE CODE

17. SECURITY CLASSIFICATION OF REPORT	18. SECURITY CLASSIFICATION OF THIS PAGE	19. SECURITY CLASSIFICATION OF ABSTRACT	20. LIMITATION OF ABSTRAC
UNCLASSIFIED	UNCLASSIFIED	UNCLASSIFIED	UL

13. Abstract (Continued)

The effect of different treatment on glass fiber on epoxy cure kinetics has been probed by evanescent mode using DDS excitation peak shift. This method is able to detect apparent differences in epoxy cure between interphases and bulk as well as after surface plasma treatment on glass fibers.
In polyimides, all the intermediate reaction species have been synthesized and characterized to investigate the kinetics and mechanisms of thermal and chemical imidization, using an extrinsic sensor of 1,5-diamino naphthalene. By combining UV, fluorescence, FTIR and GPC results, thermal imidization was thoroughly investigated in dilute solution as well as in solid state. Fluorescence was able to conclusively quantify the amount of depolymerization occurring in dilute solution.

List of All Publications:

1. Sung, C.S.P. and N.H. Sung, "UV and Emission Studies of Cure, Water and Oxidation/Degradation in Polymers", ACS Polymer Preprints, 33-1, 848, 1992.
2. Kailani, M.H., C.S.P. Sung and S.J. Huang, "Syntheses and Characterization of Model Imide Compounds and Chemical Imidization Study", Macromolecules, 25, 3751, 1992.
3. Dickinson, P. and C.S.P. Sung, "Kinetics and Mechanisms of Thermal Imidization Studies by UV-Visible and Fluorescence Spectroscopic Techniques", Macromolecules, 25, 3758, 1992.
4. Song, J. C. and C.S.P. Sung, "Fluorescence of Aromatic Diamines for Epoxy Cure Studies", ACS Polymer Preprints, 32-2, 362, 1991.
5. Paik, H. J., Sung, N. H. and C.S.P. Sung, "In-Situ Cure Monitoring of Aromatic Diamine -Epoxy System by Fiber Optic Fluorescence", ACS Polymer Preprints, 32-2, 669, 1991.
6. Dickinson, P. and C.S.P. Sung, "Kinetics of Imidization and Dissociation During Thermal Imidization", ACS Polymer Materials Science and Engineering Proceedings, 64, 129, 1991.
7. Sung, N. H., W. Dang, H. J. Paik, and C.S.P. Sung, "In-Situ Monitoring of Epoxy Cure by Fiber-Optic Molecular Sensors", SAMPE Proceedings, 1461, 1991 (San Diego, CA).
8. Pyun, E. and C.S.P. Sung, "Network Structure in Diamine-Cured Tetrafunctional Epoxy Matrix by UV-Visible and Fluorescence Spectroscopy", Macromolecules, 24, 855, 1991.
9. Dang, W., Sung, N. H. and C.S.P. Sung, "Optic Fluorophore Sensors", ACS Polym. Mat. Sci. & Eng., Proceedings, 63, 512, 1990.
10. Dickinson, P. and C.S.P. Sung, "Dissociation and Imidization Studies of Naphthyl Diamine Based Poly(Amic Acid)", ACS Polymer Preprints, 31-1, 574, 1990.
11. Kailani, M. H., Sung, C.S.P. and S. J. Huang, "Synthesis-Characterization of Model Imide Compounds and Chemical Imidization Studies", ACS Polymer Preprints, 31-1, 449, 1990.

List of All Participating Scientific Personnal:

C.S.P. Sung
J.C. Song (Ph.D. expected in 1993)
M.H. Kailani (Ph.D. expected in 1993)
P. Dickinson (Ph.D. 1990)
N.H. Sung
H.J. Paik (Ph.D. expected in 1993)
W. Dang (M.S. 1990)
A. Fuchs (Ph.D. expected in 1994)